AN INTERFERENCE MICROSCOPE STUDY OF IRON WHISKERS

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High precision measurements utilizing the interference of light waves have been widely used for many years. The combination of microscopy with interferometry enables one to obtain a three dimensional study of opaque objects in the macro and microscopic range.

Interference microscopy has proven to be an invaluable technique for the observation of nucleation habits and branching phenomena of iron whiskers. A Zeiss interference microscope ~~has been~~ was employed in an investigation having as its threefold objective: ~~first,~~ (1) the study of crystal growth preceding nucleation sites on existing whiskers; ~~second, to~~ (2) determine whether branching whiskers as well as new growth directions maintain~~ed~~ the same crystallographic continuity and, ~~third, to~~ develop a visual technique of ascertaining the degree of perfection of the crystal faces. The whiskers studied ranged in length from a few ~~millimeters~~ to ~~two centimeters~~ 2 cm.

It is possible to determine variations in surface topography from 0.03μ to 2μ. ~~These differences in heights of the surface planes can be~~ determined by the relation $t = (n)(\frac{\lambda}{2})$ where (t) is the difference in height, λ is the wavelength and n is the band deflection in fractions of the band interval. For thallium light $\lambda/2 = 0.27\mu$ and for white light the effective average $(\frac{\lambda}{2})$ is 0.3μ. The actual measurement of the various deviations was only secondary in our study. Our main concern was the utilization of the interference phenomena of light to illuminate the surface profile and thus reveal minute surface structures.

*Operated with support from the U. S. Army, Navy, and Air Force.

Initially the specimens were embedded in collodion on a glass slide but this technique precluded the remounting of the whiskers for observation on other surfaces. A punched out piece of double sticky tape, shown in Fig. 1, proved to be a more versatile mount. Also shown is another successful mounting material Stik-Tacks,* wax-like discs which allow easy reorientation of the whisker.

Although whiskers are often assumed to be perfect single crystals, variations within the field of observation (at 480X) reveals some defects in nearly all specimens investigated. Figure 2 is a grouping of three, more or less, perfect crystals. (a) is a cubic shaped whisker having a [100] growth axis and 4 $\{100\}$ surfaces, (b) is a ribbon or thin blade type growing on a [100] axis which generally is bounded by four (110) planes of extremely regular surfaces. (c) is a hexagonal shape with a [111] growth axis and has six (110) surface planes.

The terminations of whiskers if not interrupted can be classed as near perfect, Fig. 3(a). Note here that this is a blade type whisker with four [110] faces. The 55° angle of the inclined terminating plane relative to the (110) face and the $\langle 100 \rangle$ growth direction suggest that it is a (111) plane. Whenever an environmental change occurs a polycrystalline area precedes the termination, Fig. 3(b). In the case of an obstruction such as another whisker or a side wall a complete rotation of growth occurs at the band. A loss of one or more of the faces is not uncommon as the whisker terminates into an extremely fine tip which appears to be almost cylindrical, Fig. 3(c).

Observations of many whiskers under normal microscopic conditions seemed to indicate perfect or near perfect surface structure. However, using the

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*From Stik-Tack Co., Cambridge, Mass.

interference fringes to illuminate the surfaces, many interesting irregularities appear, Fig. 4. The whisker in (a) has reassumed its cubic growth after having an extensive polycrystalline area and a necking down to a cross section of only one half that of the original. An interesting phenomenon has taken place in (b) where a cubic growth has changed to a rectangular or ribbon shape thus going from four (100) faces to two (110) and two (100) faces. Complete disorder has occurred and then is followed by a very regular (110) surface. We have found the (110) faces to be mirror-like under most conditions. Whenever a whisker becomes polycrystalline there is a tendency to return to a normal or single crystal growth. This is probably because further nucleation is avoided and the total surface and grain boundary energy is lowered. In Fig. 4(c) this tendency is repeated along with several surface irregularities. Immediately following the necking down there are a series of irregular interference lines indicating depressions of approximately 0.15 microns in depth. The irregularity appears then to be resumed in the next distorted area.

It has been shown by Nabarro and Jackson[1] that whiskers grow from dislocation sites on single crystals. Certain indications of this are often seen in the formation of etch pits on strained whiskers.[2] Helical growth or growth twins are suggested when the orientation of the whisker is the same throughout, but reveal 90° or 180° twists of the growth axis when illuminated by interference fringes as shown in Fig. 5(a). These twists or rotation of the growth axis are sometimes accompanied by change in crystalline face, Fig. 5(b). Here the cubic growth characterized by four (100) faces is twisted 45° and a whisker bounded by four (110) faces results. Two consecutive 180° twists are observed following the high strain areas on the whisker in Fig. 5(c).

A whisker naturally bent under its own weight is shown in Fig. 6(a) with the broadening of interference bands resulting from the high bending strain. Such areas induce dislocations thus affording nucleation sites for the growth of new whiskers. Fig. 6(b) shows such a condition, here the new growth has assumed crystallographic continuity with the host whisker. Simple branching occurs during environmental changes such as interruption of gas flow, or fluctuations in temperature. Impurities in the combusion boat can also be a factor in this phenomenon. The base whisker shown in Fig. 6(b) is [100] direction with four (100) faces but in the overgrowth it has changed to [111] growth direction. This hexagonal region is followed by a region of high strain with subsequent growth in the original [100] direction. A near perfect [100] branch whisker has nucleated and grown from the region of high strain. Thus the energy required to support nucleation is obtained in several ways.

The dendritic-like growth in the micrograph, Fig. 6(c) was produced on a whisker from a previous reduction. This host whisker was disturbed when the boat was recharged. Its surface was then hydrogen cleaned when the new charge was brought up to reduction temperature in the hydrogen atmosphere. There was also the possibility of acid cleaning from the HCl gas that is produced when $FeCl_2 \cdot 6H_2O$ is reduced. Whenever a whisker meets a side wall or another whisker and the proper environmental conditions are present growth can continue in a new direction but with one of the bounding planes common to both segments. In Fig. 7(a) a (110) face is common to both the [100] segment and the new [112] segment as is indicated by the uniform interference bands. In Fig. 7(b) a whisker with a 90° shift in growth axis is noted while maintaining large (110) faces on both segments. In the case of the hexagonal whisker of Fig. 7(c) considerable disturbance has occurred at the termination of the original growing direction. It appears that new nucleation and growth have taken place. This is concluded because of the growth and necking down that has occurred from one of the terminating faces of the obstructed whisker.

The new type of growth that has been observed here can be further studied in Fig. 8(a) where due to a change in growth habit a perfect node has formed. The host has a [100] growth axis and a cubic form. The new whisker has started with these same characteristics. Two perfect nodes are shown in Fig. 8(b). It can be observed here that the new growth influences the entire growth habit of the existing whisker. A similar site in Fig. 8(c) has produced a whisker of the same type as the host, however the dislocation disturbance that caused the new node to start has continued along in the new whisker.

~~The~~ use of the illumination provided by the interference fringes ~~to~~ made possible to a ... the the study ~~the topography~~ of ~~the~~ metallic whiskers ~~has enabled us to fulfill the~~ threefold objective, ~~set out in the beginning of this study~~. Namely we have been able to observe nucleation sites on existing whiskers. Changes in growth axis have been determined by means of the reversal of the interference fringes. This technique is particularly useful when used in conjunction with ~~x-ray orientation~~ studies. The micrographs presented ~~have~~ demonstrate the usefulness of the interference microscope as another technique in determining crystal perfection by a visual method.

ACKNOWLEDGMENT

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2. Coleman, R. V., ibid.

FIGURE CAPTIONS

Fig. 1 Sticky tape and wax disc for mounting whiskers.

Fig. 2 Nearly perfect whiskers (a) cubic (b) rectangle (c) hexagonal.

Fig. 3 Whisker terminations (a) normal (b) polycrystalline (c) extended taper.

Fig. 4 Whiskers showing various defects (a) necking down (b) change in growth axis (c) resumption of single crystallinity following disturbed area.

Fig. 5 Whiskers showing twists (a) 90° change (b) 45° change (c) two 180° twists.

Fig. 6 Nucleation sites in whiskers (a) at bend (b) new growth from strain (c) dendritic type growth on old whisker.

Fig. 7 Growth axis changes due to collisions (a) in plane (b) continuous growth (c) from terminating plane.

Fig. 8 Nodes on iron whiskers (a) initial (b) partial (c) branch.

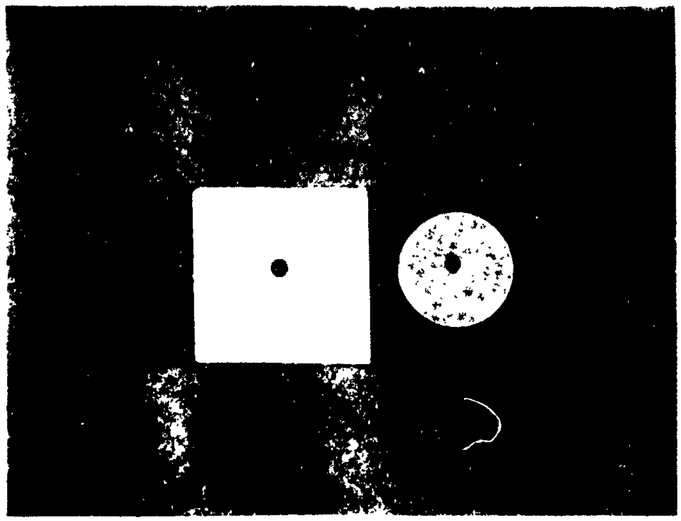
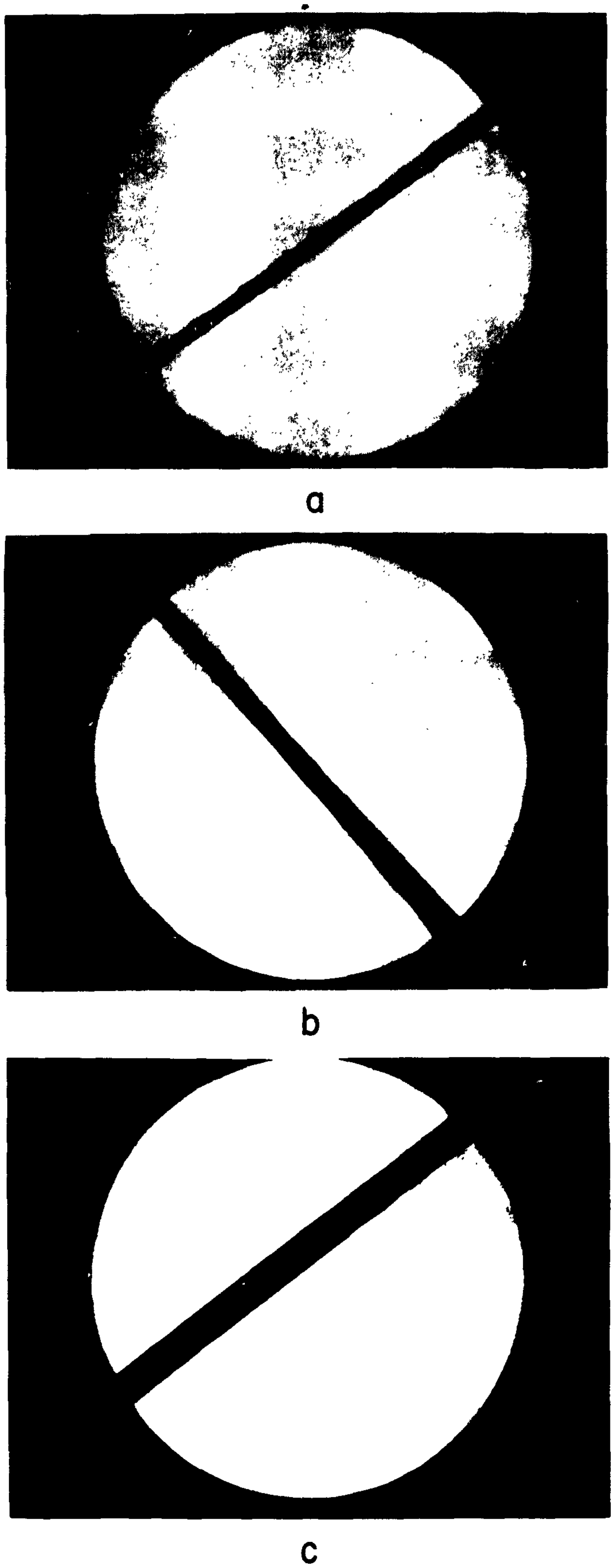


Fig. 1

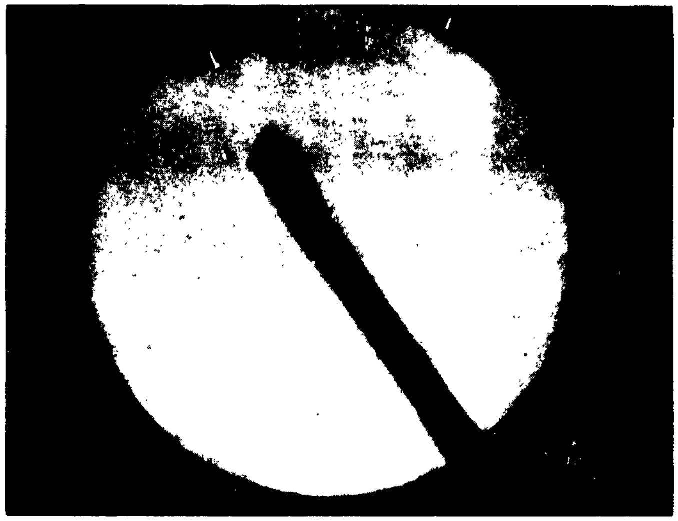


a

b

c

Fig. 2



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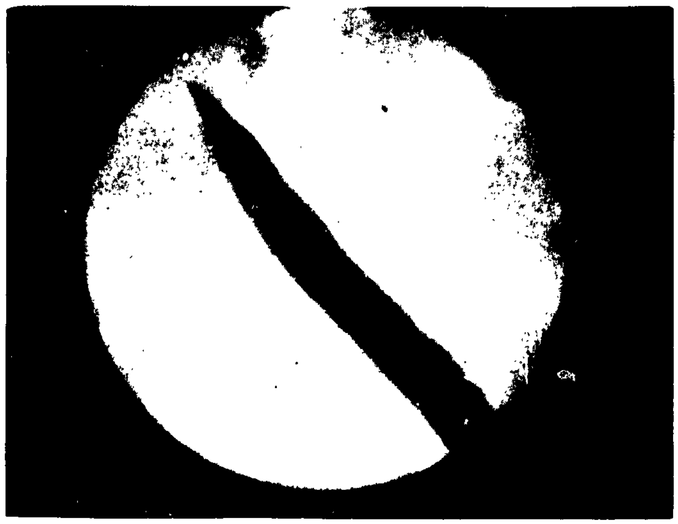

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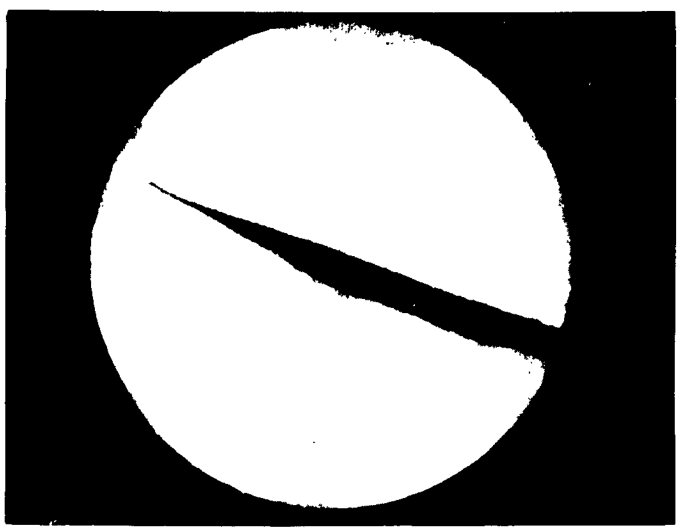


c

Fig. 3

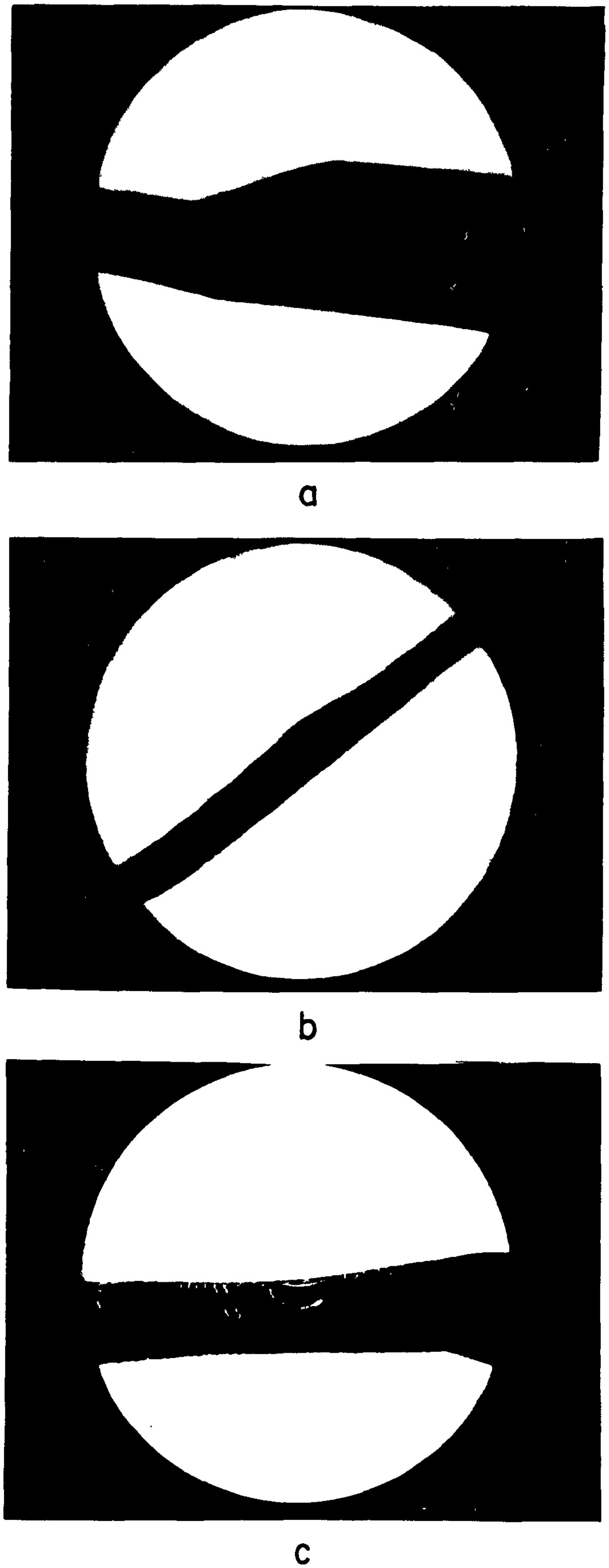
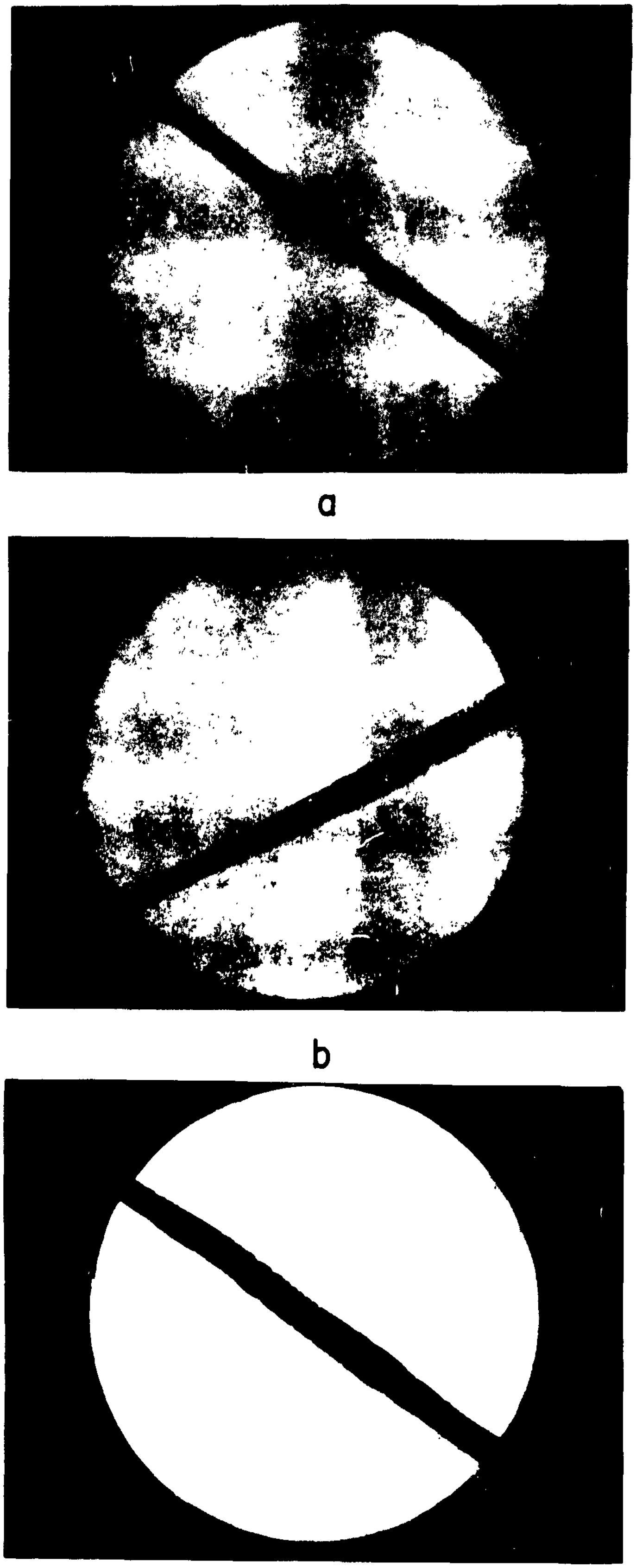


Fig. 4

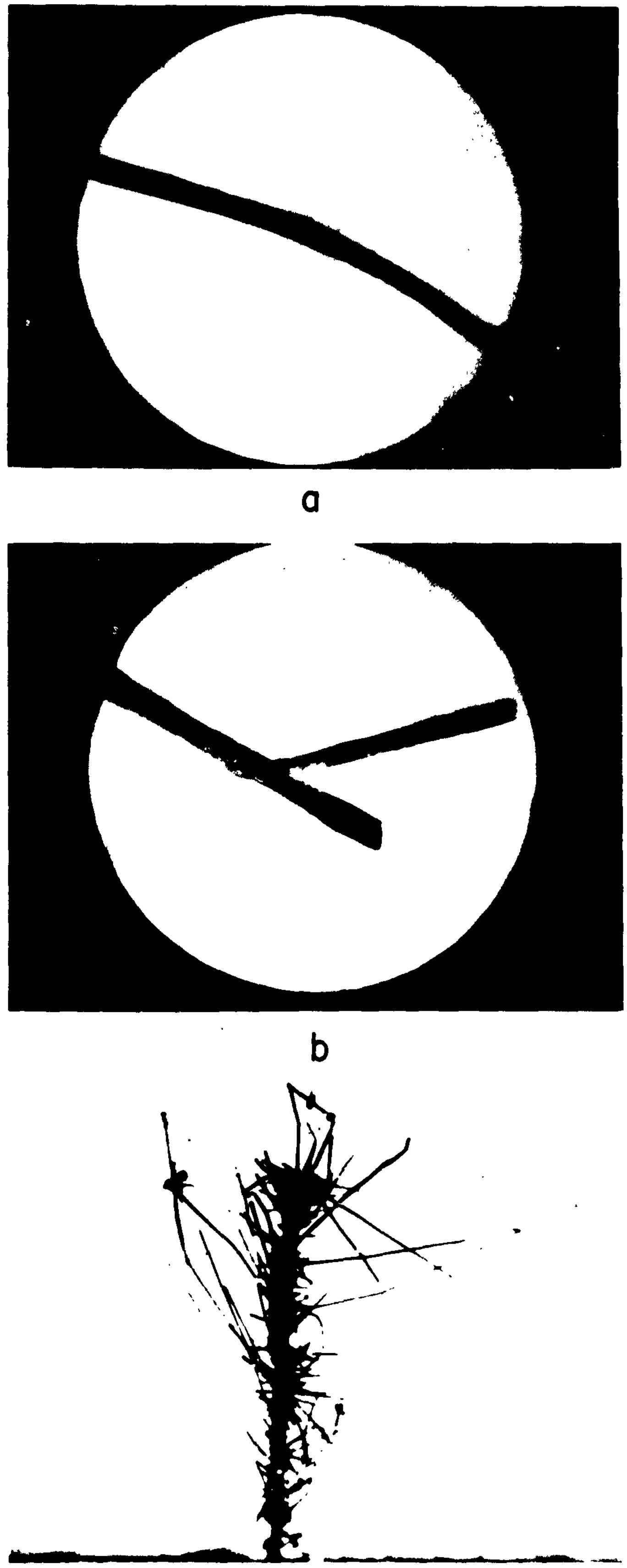


a

b

c

Fig. 5



a

b

c

Fig. 6

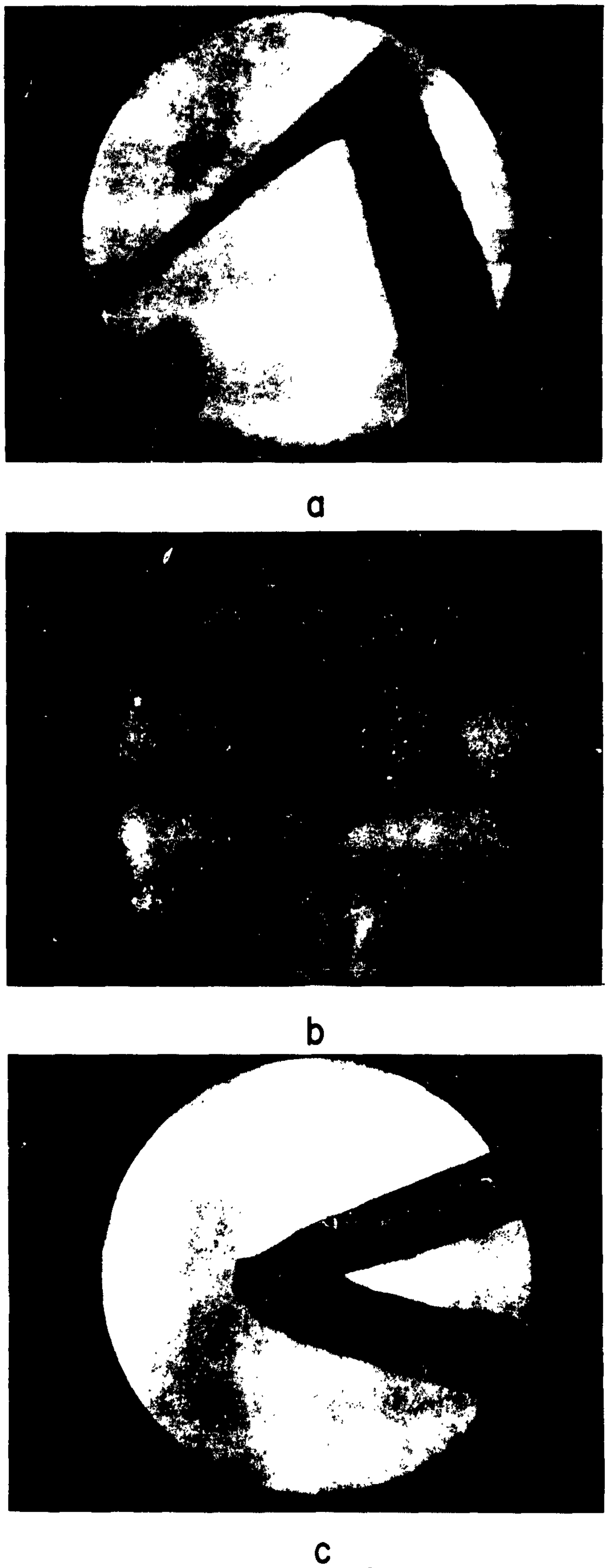
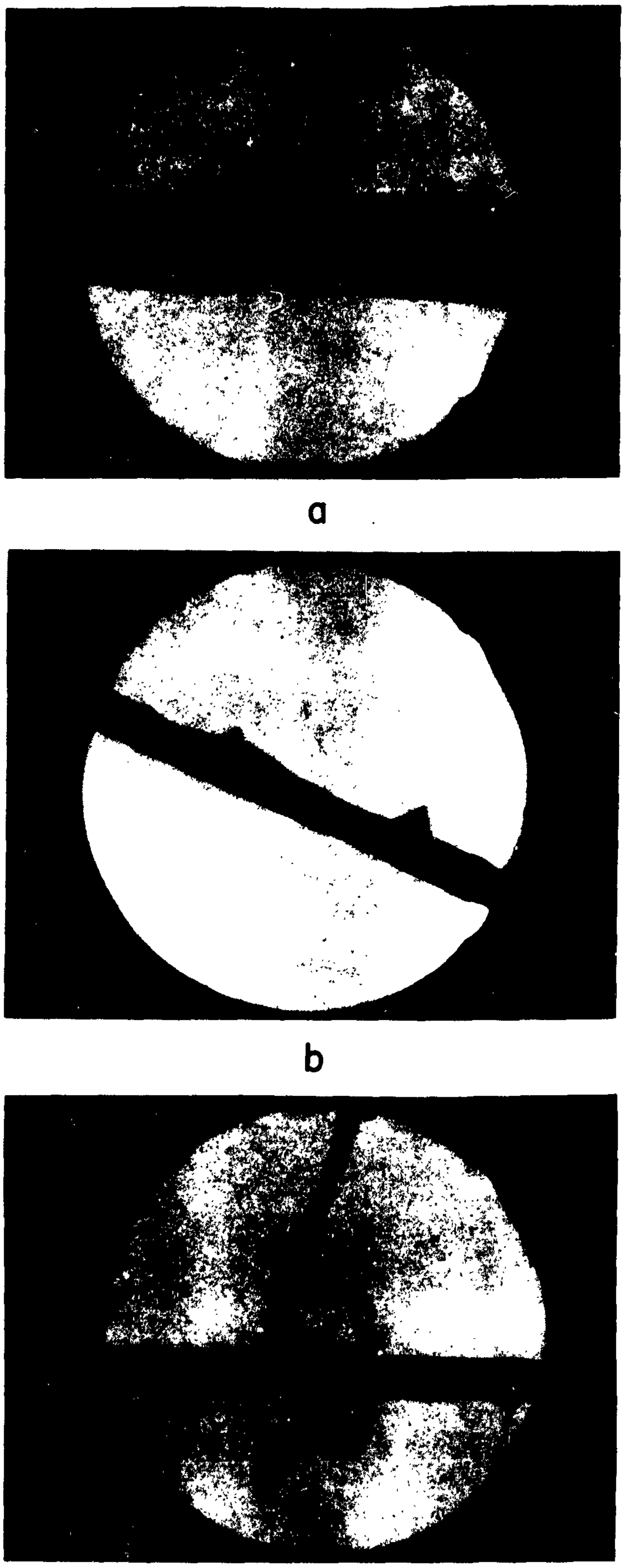


Fig. 7



a

b

c

Fig.8